AFRL-SR-BL-TR-98-

0574

REPORT DOCUMENTATION PAGE

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existir the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for r Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduc

1. AGENCY USE ONLY *(Leave blank)*	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	JULY 31, 1998	FINAL REPORT 1 MAY 97 - 30 APR 98

4. TITLE AND SUBTITLE
MICROSTRUCTURAL CHARACTERIZATION OF INTERFACES IN SIC-REINFORCED TITANIUM ALLOY-MATRIX COMPOSITES

5. FUNDING NUMBERS
F49620-97-1-0341
61102F
2306/AS

6. AUTHOR(S)
DR JONG M. K. WIEZOREK, AND DR. MICHAEL J. MILLS

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
OHIO STATE UNIVERSITY
2041 COLLEE ROAD, 477 WATTS HALL
COLUMBUS OH 43210-1179

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
AIR FORCE OFFICE OF SCIENTIFIC RESEARCH (AFOSR)
110 DUNCAN AVENUE ROOM B115
BOLLING AFB DC 20332-8050

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION AVAILABILITY STATEMENT
APPROVED FOR PUBLIC RELEASE, DISTRIBUTION IS UNLIMITED

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*
A limiting design characteristic of carbon-coated, aligned-fiber-reinforced, metal-matrix composites is the mechanical strength of the fiber/matrix interface for these composite systems. In this program, a procedure for the structural analysis of the critical carbon layer has been developed based on processing of TEM diffraction patterns or high resolution TEM images. It is demonstrated that the structural differences between various regions of the same carbon layer can be distinguished and quantified. Similar analyses should aide in the comparison of different carbon-coated, fiber-reinforced composites.

14. SUBJECT TERMS	15. NUMBER OF PAGES
	16
	16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	

Standard Form 298 (Rev. 2-89) (EG)
Prescribed by ANSI Std. 239.18
Designed using Perform Pro, WHS/DIOR, Oct 94

DTIC QUALITY INSPECTED 1

1 2 AUG 1998

Final Report on
A One Year Research Project
AFOSR Grant #F49620-97-1-0341

Microstructural Characterization of Interfaces in SiC-Reinforced Titanium Alloy-Matrix Composites

Prepared by:

Dr. Jörg M.K. Wiezorek,
Dr. Michael J. Mills

Department of Materials Science and Engineering
The Ohio State University
2041 College Road, 477 Watts Hall
Columbus, OH 43210-1179

Submitted to:

Metallic Structural Materials Program
Air Force Office of Scientific Research
Bolling Air Force Base, Building 410
Washington, D.C.
Attention: Dr.Spencer Wu

July 31, 1998

19980825 011

DTIC QUALITY INSPECTED 1

Summary

A limiting design characteristic of carbon-coated, aligned-fiber-reinforced, metal-matrix composites is the mechanical strength of the fiber/matrix interface region under transverse loading conditions. There is at present no clear correlation between the mechanical properties and the structure of the fiber/matrix interfaces for these composite systems. In this program, a procedure for the structural analysis of the critical carbon layer has been developed based on processing of TEM diffraction patterns or high resolution TEM images. It is demonstrated that the structural differences between various regions of the same carbon layer can be distinguished and quantified. Similar analyses should aide in the comparison of different carbon-coated, fiber-reinforced composites.

Introduction

The work performed at The Ohio State University (OSU) for the research project "Microstructural Characterization of Interfaces in SiC-Reinforced Titanium Alloy-matrix Composites" included the preparation of thin foils suitable for detailed characterization by transmission electron microscopy (TEM) from bulk composite samples obtained from the Wright Laboratory, Wright-Patterson Air Force Base (WPAFB), performing the TEM based microstructural characterization of these foils, and the development of strategies for quantitative data analysis and interpretation. The main objective of the research to be performed at OSU was the microstructural characterization of the interfacial region between the SiC-fiber and the Ti-alloy matrix of directionally reinforced composites. On the basis of the mechanical testing performed at WPAFB the structure and chemistry of the carbon-layers deposited on the SiC-fibers have been identified to be of specific importance for the development of structure-property relationships for these SiC-fiber reinforced Ti-6Al-4V matrix composites. Hence, during this project the structure and chemistry of these carbon-layers have been studied by advanced techniques of TEM which included imaging by conventional diffraction contrast (CTEM) and high resolution TEM (HREM), as well as diffraction methods such as selected area diffraction (SAD) and micro-diffraction (MD).

Experimental Procedures and Sample Preparation

Thin foils for the TEM studies have been prepared successfully from bulk samples of Ti-6Al-4V alloy composites containing either Textron SCS-6 fibers or Amercom Trimarc coated SiC-fibers. Electro-discharge machining was used to obtain through thickness cylinders about

3 mm in diameter form sheets of the composites . Slices about 500 μm thick were sectioned from these cylinders normal to the long cylinder-axis using a precision diamond wheel saw. The resulting 3 mm disks contained a single ply of the aligned SiC-fibers running horizontally across the full diameter of the disks. Grinding of these latter disks with abrasive paper to a thickness between 135-160 μm (the approximate fiber diameter was 140 μm) was followed by dimpling and Argon ion-milling in a Gatan Duo-Mill until perforation. Sample preparation was more successful for the composites containing Textron SCS-6 fibers. For the Amercom Trimarc SiC-fiber reinforced composites failure by fracture of the fibers or the whole thin disks occurred frequently, thus preventing successful TEM foil preparation. The TEM studies focused on these later samples. The experimental TEM work was performed using a Philips CM300FEG/UT operated at 300 kV, capable of spatial resolution of detail down to about 1.1 Å in HREM mode, and equipped with a Gatan Multiscan digital camera for image pick-up as well as a light element energy dispersive X-ray (EDS) detector and a digital parallel electron energy-loss spectrometer (PEELS) for chemical analysis. Diffraction and imaging data have been collected both on the conventional photographic plate media and also digitally using the Multiscan camera. The digital analyses of the HREM images and the SAD was performed by using the commercial software packages SEMPER and MacTempas.

TEM Observations of the Carbon layers

Overview

All the TEM observations presented in this report have been obtained from Ti-6Al-4V composites containing Textron SCS-6 fibers. Figure 1 shows plan view bright field (BF) TEM micrographs of the carbon-layer near the interface with the SiC-fiber at two different locations together with the corresponding SAD. The significant arcing of the strong 0002-reflections of the carbon clearly indicate considerable alignment of the basal plane layers which is typical of graphitic structures. As previously reported (Oberlin 1989, Ning and Pirouz 1991, Ning et al. 1990), a basic structural unit (BSU), an elemental building block, can be defined to describe the morphology of non-diamond-like forms of carbon. Obtaining access to a quantification of the differences in the BSU characteristics of the carbon-layers would be extremely useful to identify structure-property relationships for these composites. Information regarding the average size, shape and level of alignment of the BSU within different regions of the carbon-layers in the composites can in principle be obtained from SAD and also HREM images. The development of tools to facilitate the measurement and quantification of this microstructural information from SAD and/or HREM data was one goal of this project and will be addressed later in the report.

HREM

HREM images have been obtained from different locations within the carbon-layer, i.e. close to the interface between the Ti-matrix and the carbon, closer to the interface with the SiC-fiber, and in closest vicinity to the interface with the SiC-fiber. Examples of such HREM images are displayed in figures 2-4 together with the corresponding Fast-Fourier-Transforms (FFT) (figures 2b, 3b and 4b), the equivalent of optical diffractograms. Additionally, details of the carbon microstructure are shown at higher magnification (figures 2c, 3c and 4c). The interesting feature to note is the absence of arcing associated with spatial frequencies corresponding to the 0002-lattice spacings in the carbonaceous BSU in the FFT of the HREM image obtained from the perfectly amorphous region near the Ti-matrix carbon-layer interface (figure 2), whereas significant arcing of corresponding to these 0002-reflections is present in the FFT's of the HREM images obtained from regions closer to the interface with the SiC-fiber (figure 3 and 4). As mentioned earlier, the morphology of the arcing of the 0002-reflections is associated with the size, size-distribution, and level of alignment of the BSU's contained in the imaged area of the carbon-layer. The considerable level of alignment of the basal planes in a graphite-like fashion is most strikingly obvious in the HREM detail shown in figure 4c. The arcing of the 0002-reflection in the corresponding FFT (figure 4b) is confined to a smaller angular range than in the FFT of the data presented in figure 3. This is consistent with an increasing level of alignment of the graphitic-like basal planes and presumably corresponds to an increased size of the relevant BSU. The HREM images of figures 2-4 have been obtained from locations at different distances from the SiC-fiber carbon interface. Based on the data presented in figures 2-4 it appears reasonable to conclude that the level of alignment and probably also the mean size of the BSU increases with decreasing distance from the SiC-fibers.

Quantification of the microstructural information obtained by HREM imaging and image analysis regarding the BSU that constitute the carbon-layer would be most useful for the development of structure-mechanical property relationships for the SiC-fiber reinforced Ti-alloy matrix composites. Advantages of exploring the analysis of HREM images and the corresponding diffractograms, such as obtaining a visual picture of the lattice-fringes and the possibility of forming pseudo-dark field HREM images by appropriate masking in the diffractogram and inverse FFT (see example of this procedure presented in figure 5), are off-set by significant problems associated with the low signal-to-noise ratio inherent to diffractograms as compared to SAD. Furthermore, great care has to be taken when analyzing diffractograms to separate the artifacts introduced to the diffractogram from the contrast transfer function (CTF) of the TEM microscope. The presence of CTF information in the diffractograms would be most detrimental when it occurs at spatial frequencies that correspond to those of the 0002-lattice fringes of the carbon-layers. As a result of all these latter potential problems, which place stringent requirements on the experimental procedures when collecting useful data, it was decided not to explore further the quantitative analysis of HREM data.

Selected Area Diffraction

The schematics presented in figure 6 illustrate the strategy followed to develop tools for the quantification of the information contained in SAD patterns regarding the microstructural morphology and topology of the carbon-layers. Digital SAD patterns were used to obtain quantitative measurements of variables related to the mean size of the BSU, i.e. the radial width of the 0002-reflection arcs, $\Delta\mathbf{r}$, and the relative level of alignment of the BSU's contained in the diffraction area, i.e. the angular or azimuthal extent of the 0002-reflection arcs, $\Delta\phi$. The aim is then to generate graphic representations of the radial and the azimuthal intensity distributions, $\mathbf{I(r)}$ and $\mathbf{I}(\phi)$, in the SAD and compare quantifiable numbers, such as the full-width at half maximum (FWHM), actual maximum peak heights and sum-peak intensity, for SAD's from different regions of the carbon-layers and for different composites. Clearly useful experimental data must not contain saturated intensity in the 0002-reflections as shown as an example of unsuitable data in figure 7. An approach to successful background subtraction is schematically represented in the diagram shown in figure 8. Following such procedures for the analysis of the digital SAD patterns then facilitates the quantification of for instance the FWHM in the radial intensity distribution through the center of the 0002-reflections associated with the aligned graphitic-like BSU's in the carbon-layers.

Examples of SAD Analyses

Figure 9 and 10 collate examples of some preliminary analyses of SAD patterns. Figure 9 shows some original digital SAD plotted to the same scale, alongside the same images of the SAD with azimutal angle and radius r along the abscissa and ordinate respectively. The appropriate back-ground subtracted radial and azimuthal intensity line profiles were obtained simply by extracting horizontal and vertical sections from the images shown in the far right of figure 9. The images used had been multiplied by the radial distance r because this ensures that the intensity scattered to all angles is then correct. The radial line profiles through the center of the 0002-arcs shown in figure 10 were obtained by averaging over a narrow angular range (2.5°) and subtracting the background intensity (obtained from line profiles at 90° to the former). The table shows some numbers extracted from these line profiles, averaged over two arcs in each case. Noticeably only the profile associated with SAD160 has a substantially different FWHM from the other two profiles, while the variation in the area underneath the arcs is larger than the variation in the FWHM. Figure 10 also shows line profiles obtained through the center of the arcs but as a function of angle. These angular intensity line profiles were obtained after averaging over a small distance of 1.140 of the distance between the transmitted beam and the SiC-reflections, and are of course also back-ground subtracted. Notably and angular interval of 720° is displayed in each profile in order to display both the 0002- and the $000\bar{2}$-arc. Again in the corresponding table some numbers extracted from the angular line profiles are shown. Interestingly, the FWHM is smaller in SAD142 than for SAD160. Thus, it has been demonstrated that it is feasible to extract quantifiable information from digital SAD that relates to the microstructural morphology and topology associated with the characteristics of the BSU contained in the carbon-layers. Program codes in the SEMPER programming language can be obtained upon request with detailed comments on the functions of the routines employed.

References

Oberlin, A., Chemistry and Physics of Carbon, ed. P.A. Thrower (Marcel Dekker: New York), (1989).

Ning, X.J, and Pirouz, P., J. Mater. Res. Vol. 6, (1991), 2234.

Ning, X.J, Pirouz, P.,, Lagerlof, K.P.D., and DiCarlo, J., J. Mater. Res. Vol. 5, (1990), 2865.

PERSONNEL SUPPORTED

Funding for this year-long program has been utilized in partial support for Dr. Jorg Wiezorek's salary, as well as to offset the user fees for the TEM work. The assistance of Dr. Rafal Dunin-Borkowski at Arizona State University in thedevelopment of the SEMPER-based processing techniques is also gratefully acknowledged.

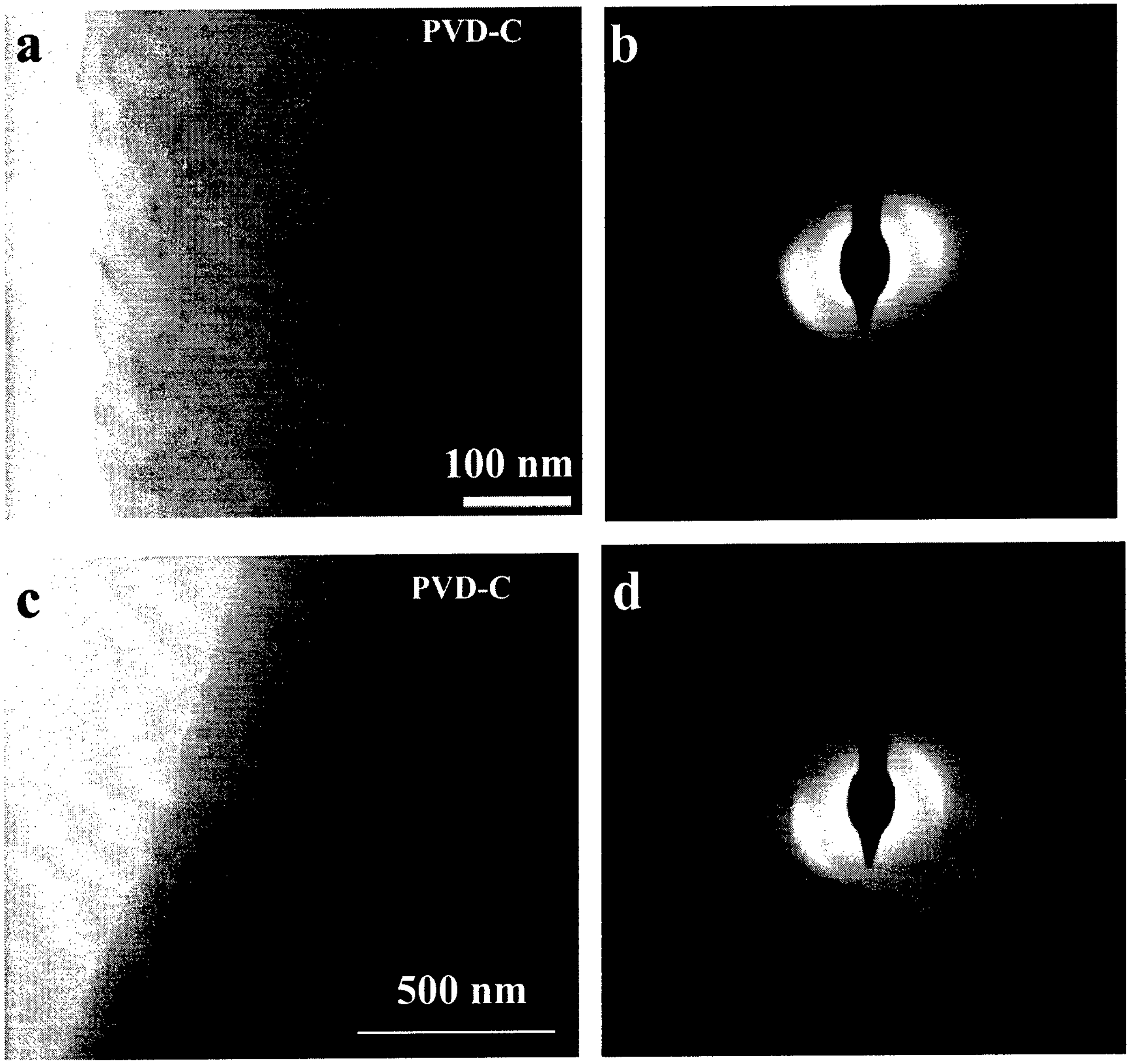


Figure 1

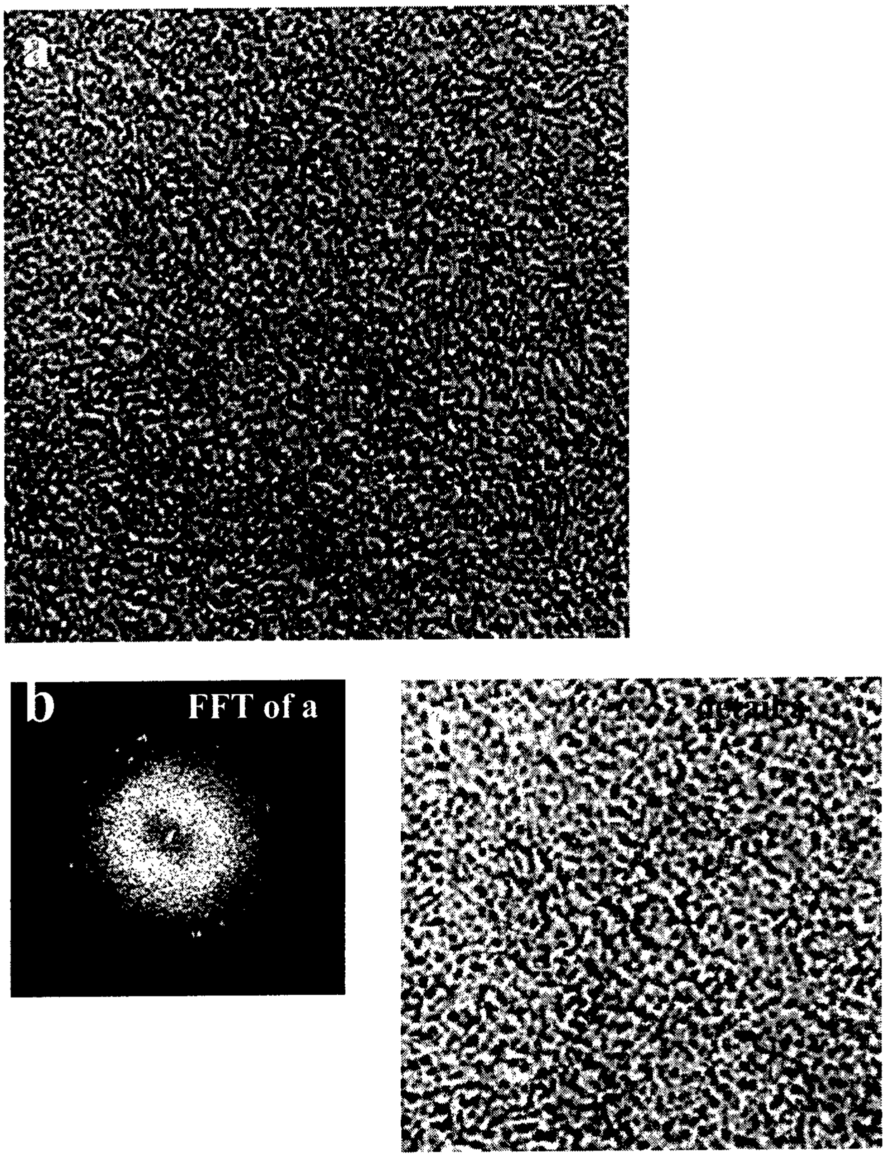


Figure 2:
Note the absence of arking in the FFT and the amorphous appearance of the PVD-carbon layer in the vicinity of the Ti-matrix for the SCS-6-fiber.

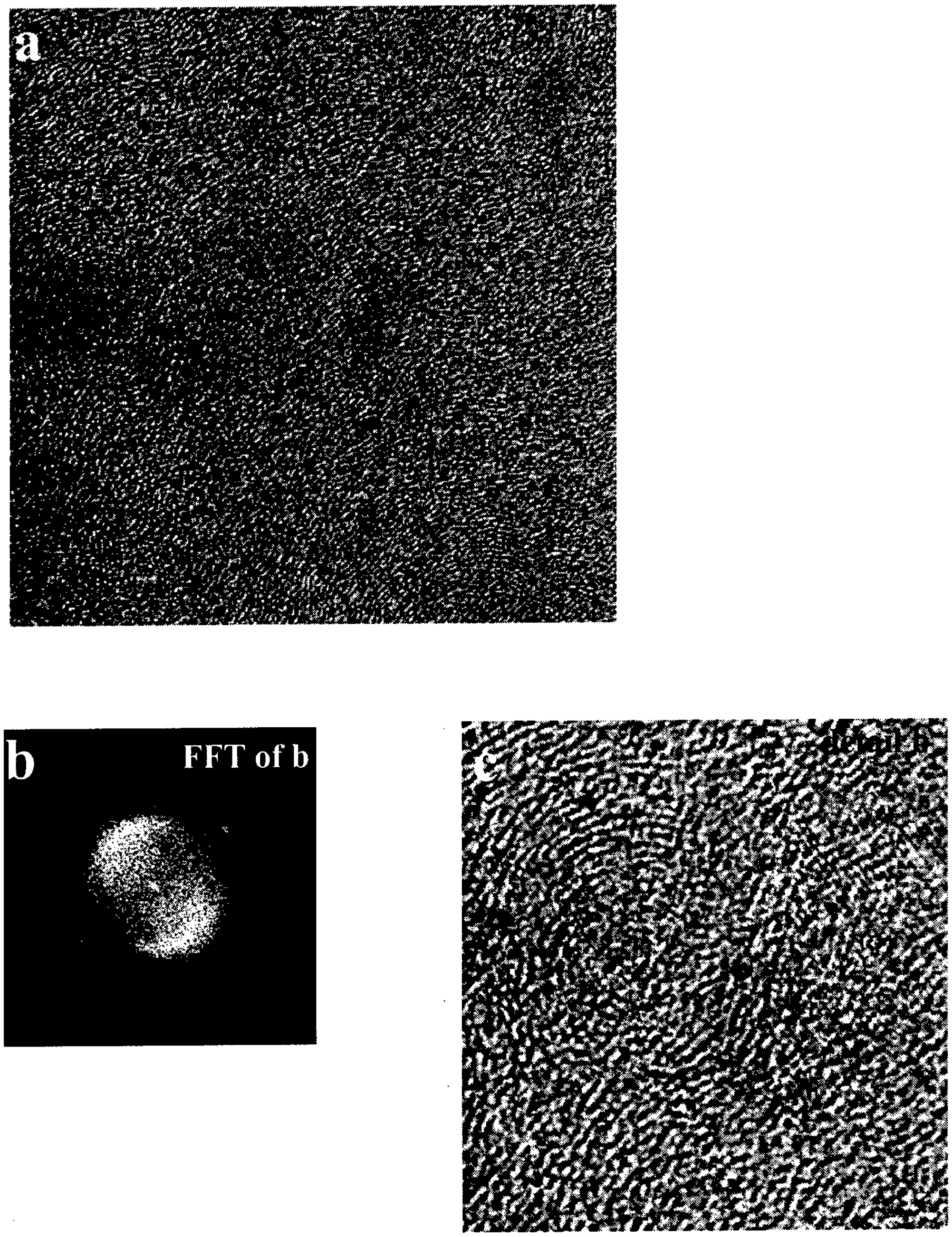
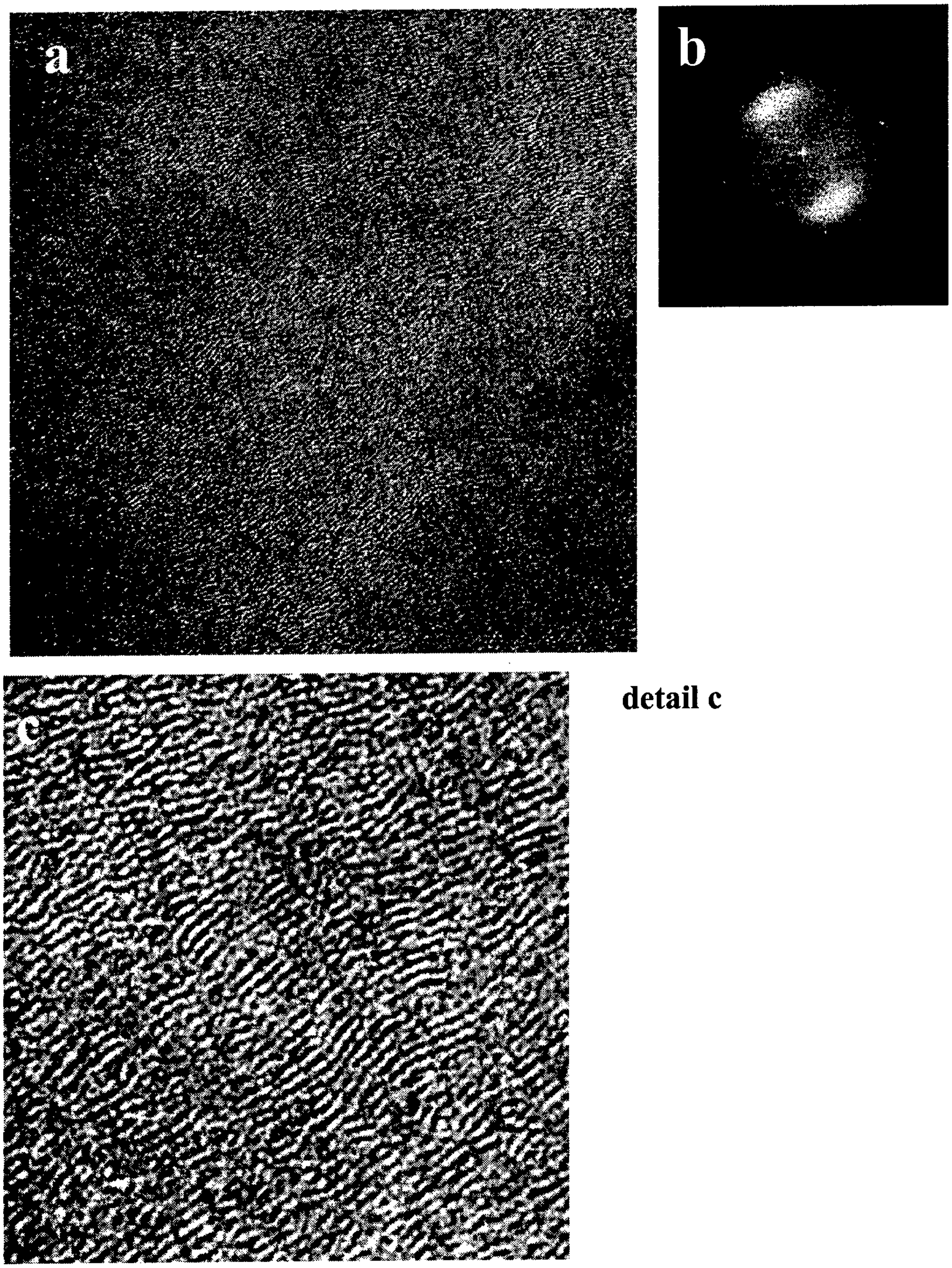


Figure 3:
Note the arking in the FFT and the turbostratic appearance with circular symmetries of the PVD-carbon layer in the vicinity of the the SCS-6-fiber.



FFT of c

detail c

Figure 4:
Note the arking in the FFT and the turbostratic appearance with near graphitic alignments of the PVD-carbon layer in the vicinity of the the SCS-6-fiber.

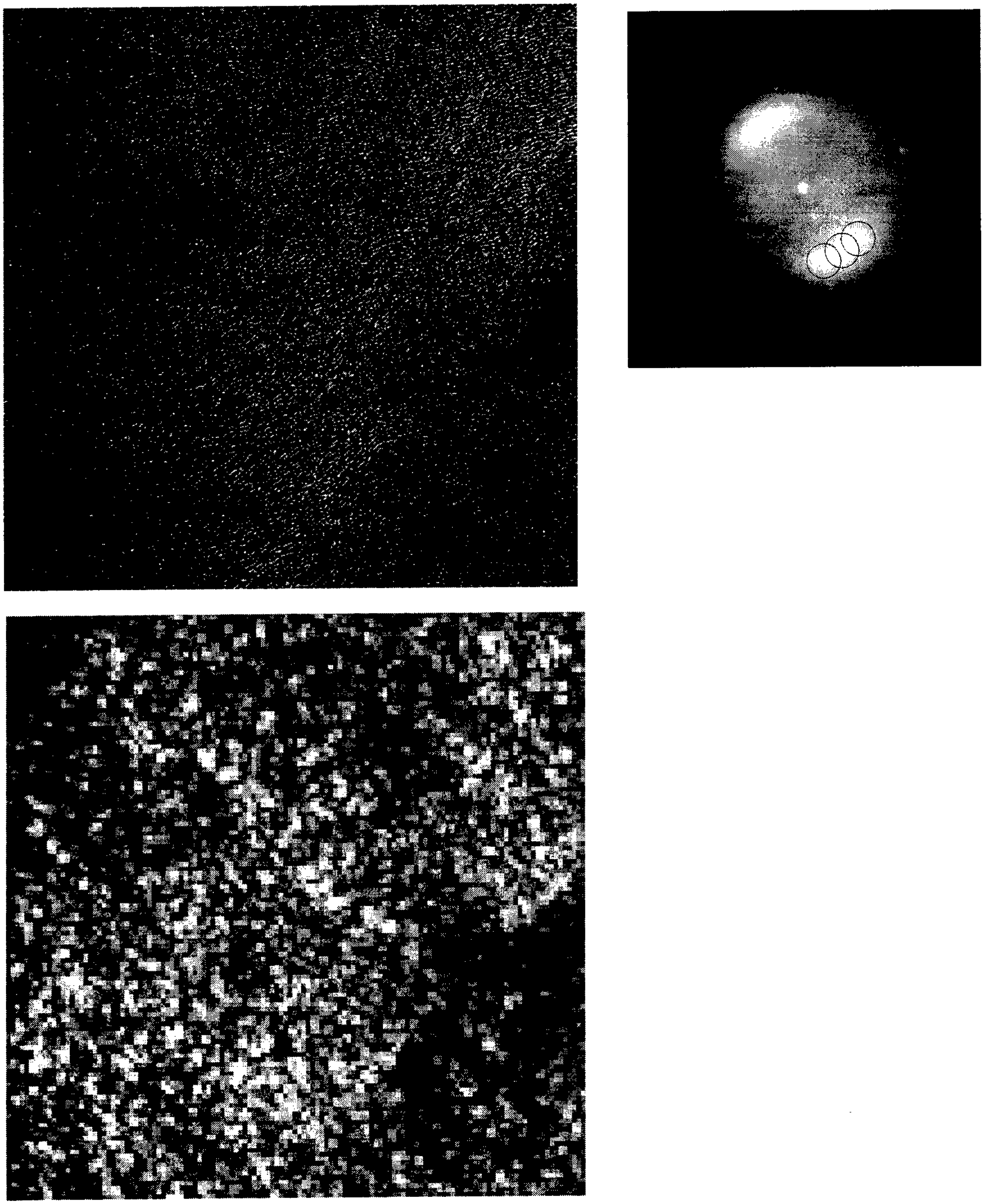


Figure 5: HREM micrograph with corresponding diffractogram, which is plotted on a log-scale to enhance contrast and indicates three location of a mask position used to obtain a pseudo-darkfield image. The three pseudo dark-field images are shown combined in the color imgagfe, Same angles of scatter, i.e alignement, correspond to the same color.

Schematic Representation of the variables or quantities to be extracted from and SAD.

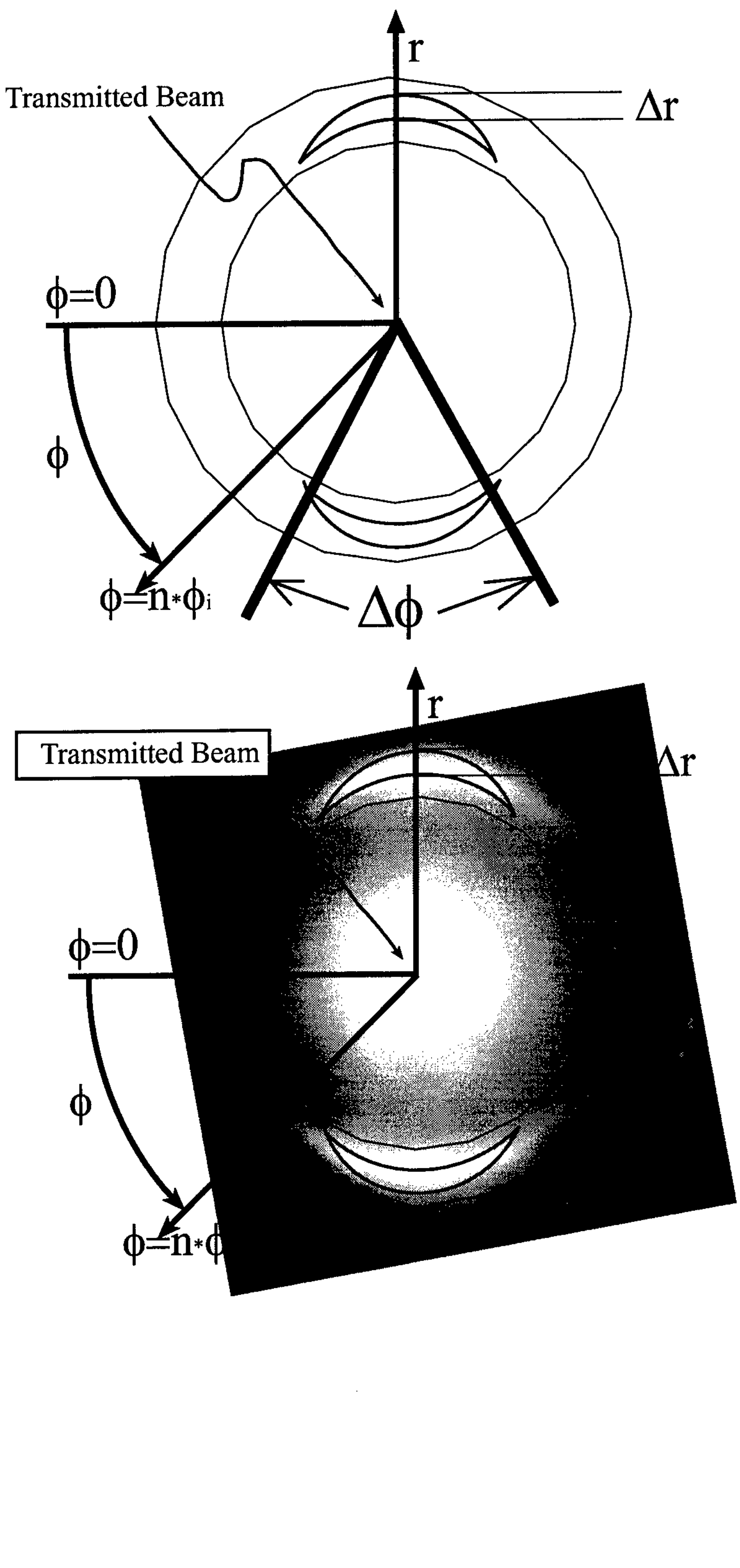


Figure 6:

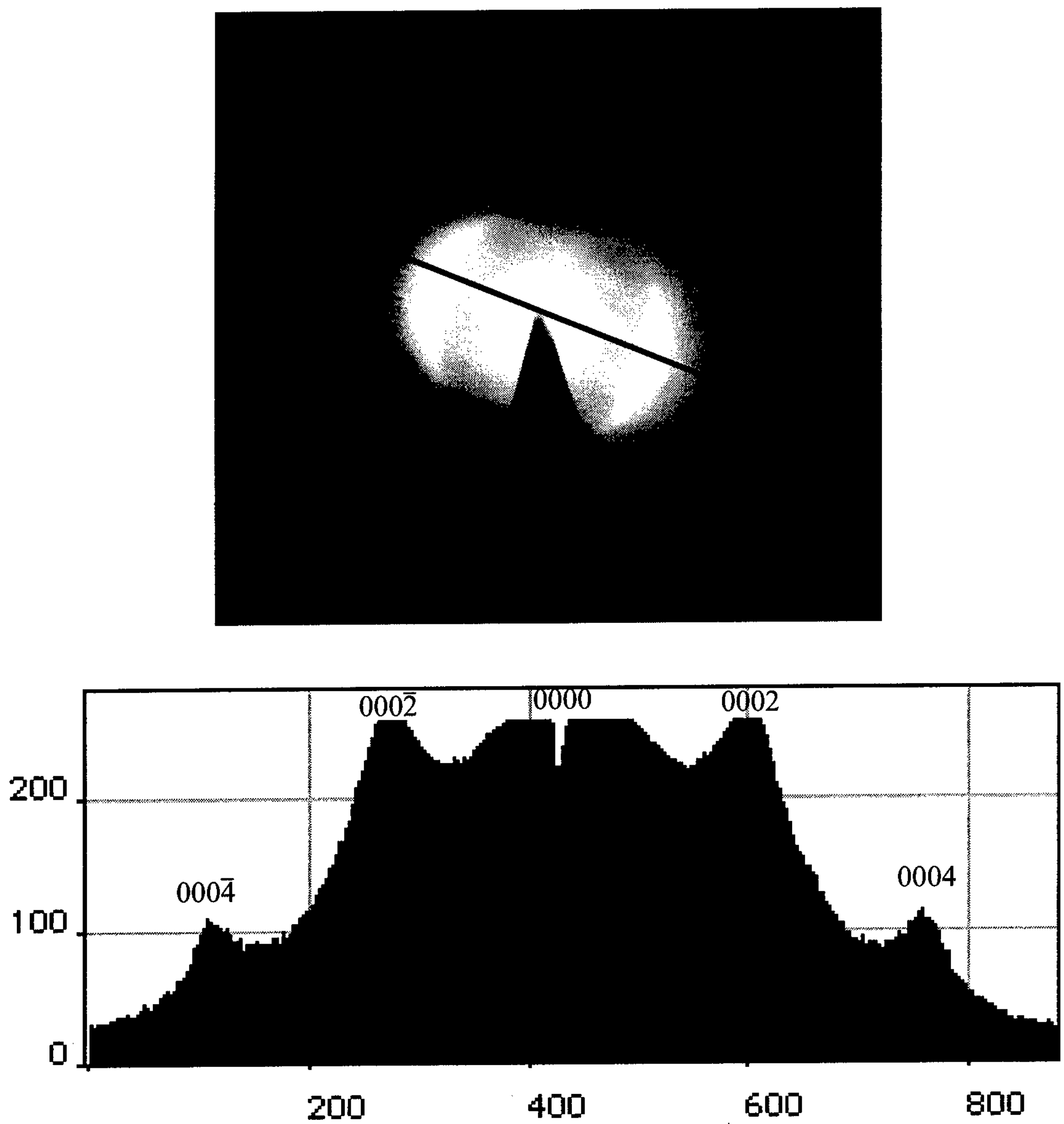


Figure 7:

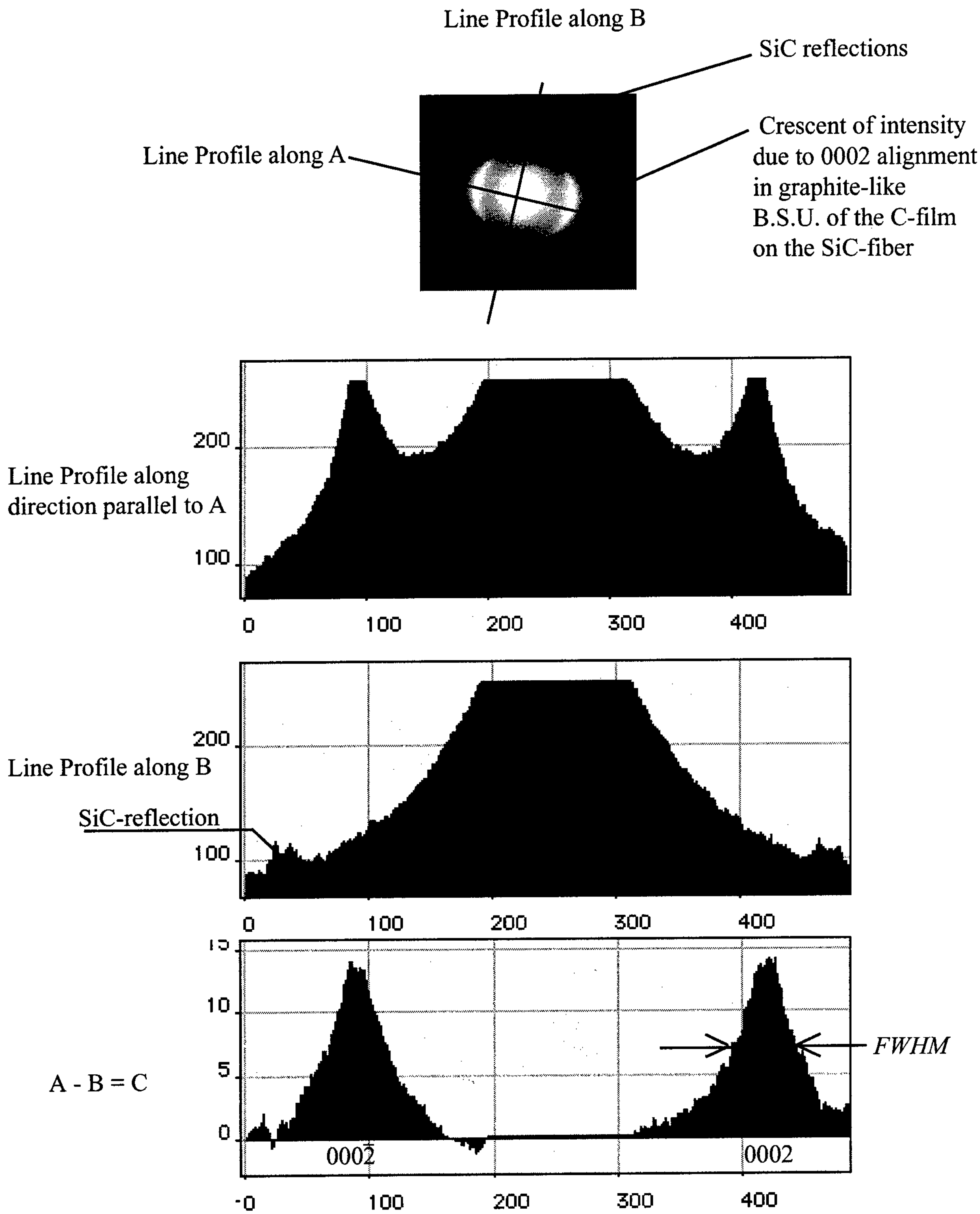
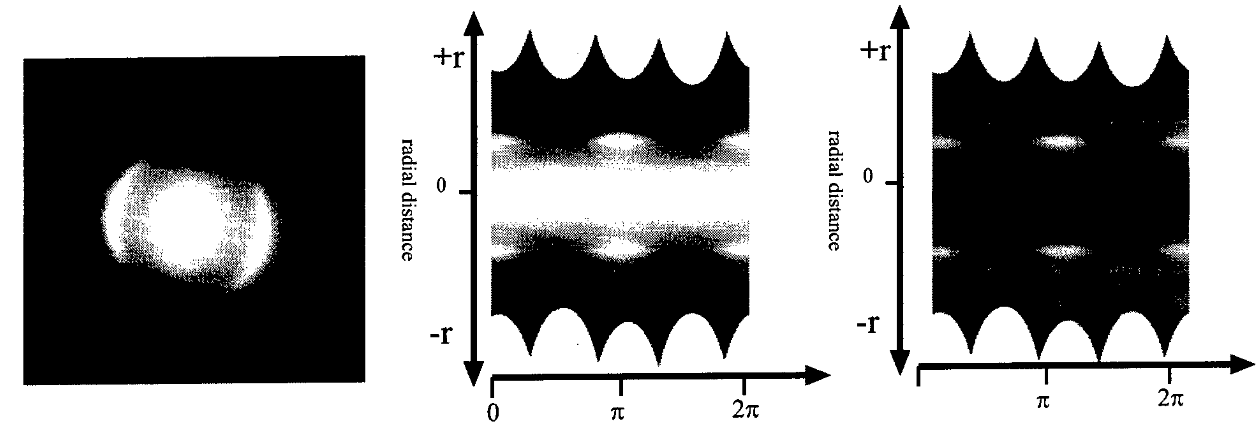
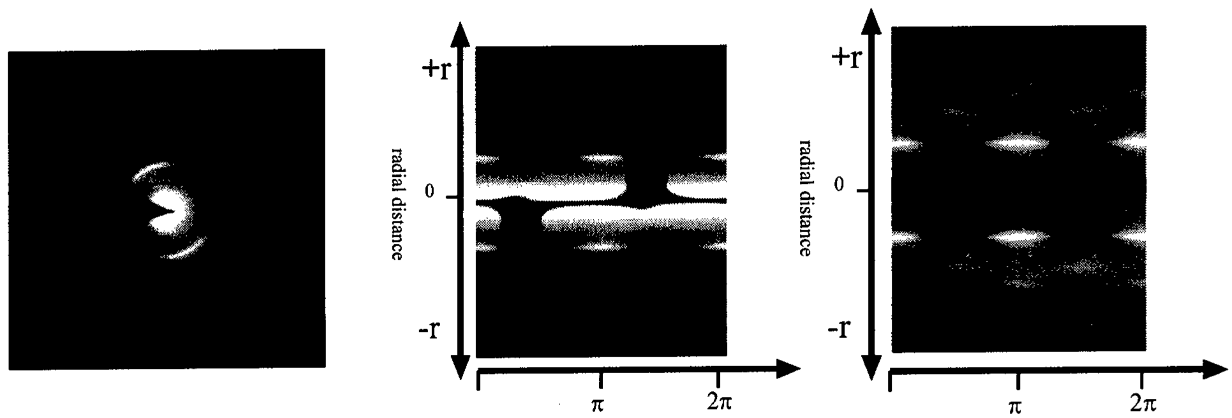

Line Profile along B
SiC reflections
Line Profile along A
Crescent of intensity due to 0002 alignment in graphite-like B.S.U. of the C-film on the SiC-fiber
Line Profile along direction parallel to A
200
100
0
100
200
300
400
Line Profile along B
SiC-reflection
200
100
0
100
200
300
400
A - B = C
15
10
5
0
FWHM
000$\bar{2}$
0002
-0
100
200
300
400

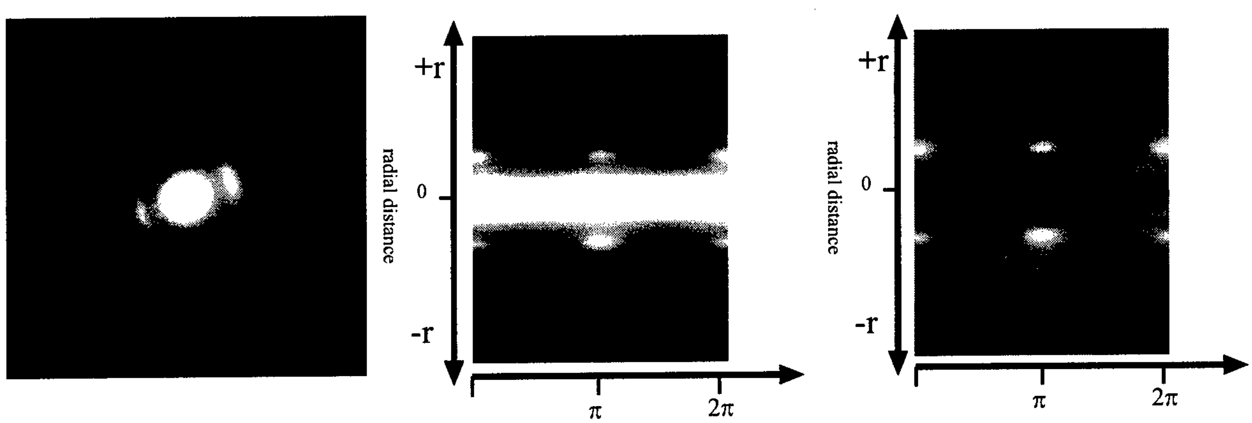
Figure 8:



SAD2



SAD142



SAD160

Figure 9:

Radial Line Profiles

Angular Line Profiles

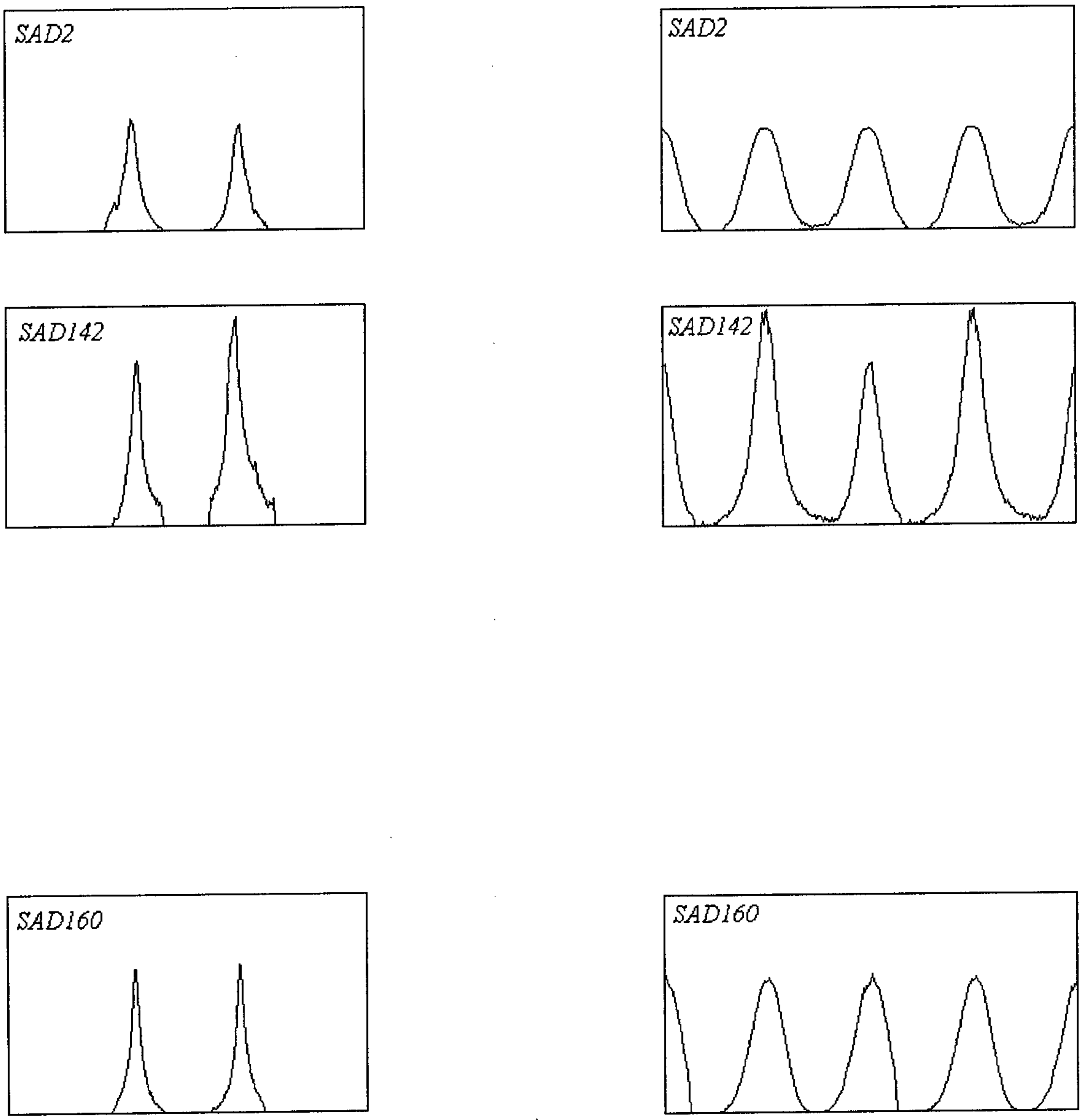


Figure 10